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### Strong competition between the delta I and delta T-c flux pinning mechanisms in MgB<sub>2</sub> doped with carbon containing compounds

Shaban R. Ghorbani

*University of Wollongong, ghorbani@uow.edu.au*

Xiaolin Wang

*University of Wollongong, xiaolin@uow.edu.au*

Md S. Hossain

*University of Wollongong, shahriar@uow.edu.au*

Qiwen Yao

*University of Wollongong & National Institute for Materials Science, Japan, qy75@uow.edu.au*

S X. Dou

*University of Wollongong, shi@uow.edu.au*

*See next page for additional authors*

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# Strong competition between the $\delta l$ and $\delta T_c$ flux pinning mechanisms in MgB<sub>2</sub> doped with carbon containing compounds

## Abstract

The transport and magnetic properties of 10 wt % malic acid and 5 wt % nanocarbon doped MgB<sub>2</sub> have been studied by measuring the resistivity ( $\rho$ ), critical current density ( $j_c$ ), connectivity factor ( $A_F$ ), irreversibility field ( $H_{irr}$ ), and upper critical field ( $H_{c2}$ ). The pinning mechanisms are studied in terms of the collective pinning model. It was found that both mean free path ( $\delta l$ ) and critical temperature ( $\delta T_c$ ) pinning mechanisms coexist in both doped MgB<sub>2</sub>. For both the malic acid and nanocarbon doped samples, the temperature dependence of the crossover field, which separates the single vortex and the small bundle pinning regime,  $B_{sb}(T)$ , shows that the  $\delta l$  pinning mechanism is dominant for temperatures up to  $t(T/T_c)=0.7$  but the  $\delta T_c$  pinning mechanism is dominant for  $t > 0.7$ . This tendency of coexistence of the  $\delta l$  and the  $\delta T_c$  pinning mechanism is in strong contrast with the pure MgB<sub>2</sub>, in which the  $\delta T_c$  pinning mechanism is dominant over a wide temperature range below  $T_c$ . It was also observed that the connectivity factor, active cross-sectional area fraction ( $A_F$ ), are 0.11 and 0.14 for the nanocarbon and the malic acid doped MgB<sub>2</sub>, respectively, indicating that there are still rooms for further improving  $j_c$  performance.

## Keywords

Strong, competition, between, delta, delta, flux, pinning, mechanisms, MgB<sub>2</sub>, doped, carbon, containing, compounds

## Disciplines

Engineering | Physical Sciences and Mathematics

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## Authors

Shaban R. Ghorbani, Xiaolin Wang, Md S. Hossain, Qiwen Yao, S X. Dou, Sung-Ik Lee, K C. Chung, and Y K. Kim

# Strong competition between the $\delta l$ and $\delta T_c$ flux pinning mechanisms in $\text{MgB}_2$ doped with carbon containing compounds

S. R. Ghorbani,<sup>1,2</sup> Xiao-Lin Wang,<sup>1,a)</sup> M. S. A. Hossain,<sup>1</sup> Q. W. Yao,<sup>1</sup> S. X. Dou,<sup>1</sup> Sung-Ik Lee,<sup>3,b)</sup> K. C. Chung,<sup>4</sup> and Y. K. Kim<sup>4</sup>

<sup>1</sup>*Institute for Superconducting and Electronic Materials, University of Wollongong, Wollongong, New South Wales 2522, Australia*

<sup>2</sup>*Department of Physics, Sabzevar Tarbiat Moallem University, P.O. Box 397, Sabzevar, Iran*

<sup>3</sup>*National Creative Research Initiative Center for Superconductivity, Department of Physics, Sogang University, Seoul 121-742, Republic of Korea*

<sup>4</sup>*Nano-functional Materials Group, Korea Institute of Materials Science, Changwon, Republic of Korea*

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The transport and magnetic properties of 10 wt % malic acid and 5 wt % nanocarbon doped  $\text{MgB}_2$  have been studied by measuring the resistivity ( $\rho$ ), critical current density ( $j_c$ ), connectivity factor ( $A_F$ ), irreversibility field ( $H_{irr}$ ), and upper critical field ( $H_{c2}$ ). The pinning mechanisms are studied in terms of the collective pinning model. It was found that both mean free path ( $\delta l$ ) and critical temperature ( $\delta T_c$ ) pinning mechanisms coexist in both doped  $\text{MgB}_2$ . For both the malic acid and nanocarbon doped samples, the temperature dependence of the crossover field, which separates the single vortex and the small bundle pinning regime,  $B_{sb}(T)$ , shows that the  $\delta l$  pinning mechanism is dominant for temperatures up to  $t(T/T_c)=0.7$  but the  $\delta T_c$  pinning mechanism is dominant for  $t > 0.7$ . This tendency of coexistence of the  $\delta l$  and the  $\delta T_c$  pinning mechanism is in strong contrast with the pure  $\text{MgB}_2$ , in which the  $\delta T_c$  pinning mechanism is dominant over a wide temperature range below  $T_c$ . It was also observed that the connectivity factor, active cross-sectional area fraction ( $A_F$ ), are 0.11 and 0.14 for the nanocarbon and the malic acid doped  $\text{MgB}_2$ , respectively, indicating that there are still rooms for further improving  $j_c$  performance. © 2010 American Institute of Physics. [doi:10.1063/1.3366710]

## I. INTRODUCTION

Enhancement of the critical current density has been a central topic of research since the discovery of superconductivity in magnesium diboride ( $\text{MgB}_2$ ), where the critical temperature ( $T_c$ )  $\approx 39$  K.<sup>1</sup> High critical current density values of  $10^5$ – $10^6$  A/cm<sup>2</sup> have been reported for this superconductor. However, the critical current density ( $j_c$ ) drops rapidly with increasing temperature and magnetic field. The  $j_c$  is controlled by many parameters, e.g., the irreversibility field ( $H_{irr}$ ), the upper critical field ( $H_{c2}$ ), the flux pinning, and the connectivity. There are two ways to improve  $j_c$ . The first one is the improvement of the intrinsic properties, such as the irreversibility field  $H_{irr}$  and the upper critical field  $H_{c2}$ . The second involves increasing  $j_c$  through improvement of extrinsic properties, such as by decreasing porosity and increasing connectivity. The research toward improvement of  $j_c$  properties in  $\text{MgB}_2$  has been mostly aimed at enhancing the  $H_{irr}$ , the flux pinning ability, and  $H_{c2}$  by irradiation or chemical doping.<sup>2–8</sup> Chemical doping has been proved to be a simple and effective way to introduce pinning centers into  $\text{MgB}_2$  superconductor or to improve the upper critical field. It is believed that carbon containing dopants, such as nano-SiC,<sup>5</sup> carbon nanotubes,<sup>6</sup> nanocarbon,<sup>7</sup> hydrocarbons,<sup>8</sup>

silicone oil,<sup>9,10</sup> malic acid,<sup>11</sup> etc., are especially effective in enhancing the  $H_{c2}$ , and hence the  $j_c$  properties of  $\text{MgB}_2$  in high magnetic fields.

Intergrain boundary pinning<sup>12</sup> and point defect pinning<sup>13</sup> are the two main important pinning mechanisms used to improve  $j_c$ . In high- $\kappa$  type-II superconductors, where  $\kappa$  is the Ginzburg–Landau parameter, the most important elementary interaction between the vortices and the pinning centers is the core interaction. It arises from the influence of the parts of the material with weak superconducting properties on the spatial variation in the superconducting order parameter. The core interaction that is generally prevalent is magnetic in nature and related to the presence of interfaces parallel to the external field between superconducting and nonsuperconducting regions. It has been found that in  $\text{MgB}_2$  bulk and thin film samples, with  $\kappa$  larger than 20,<sup>14,15</sup> the core pinning is related to randomly distributed spatial variations in the transition temperature ( $\delta T_c$  pinning).<sup>16,17</sup> However, in the high-temperature superconductors, the core interaction is associated with charge-carrier mean free path variations ( $\delta l$  pinning), mostly due to crystal lattice defects.<sup>18</sup>

The critical current density of  $\text{MgB}_2$  has been improved by more than one order of magnitude, especially in high magnetic field by adding SiC.<sup>5</sup> It has also been found that maximum flux pinning forces are increased by decreasing grain size.<sup>19</sup> Therefore, the size of the doping is particularly important in doped  $\text{MgB}_2$  samples. It is effective to reduce the grain size of  $\text{MgB}_2$  by chemical doping approach, such as nano-SiC, nano-C, or nano-Si, or using liquid precursors,

<sup>a)</sup>Electronic mail: xiaolin@uow.edu.au.

<sup>b)</sup>Prof. Sung-Ik Lee, one of our original corresponding authors, sadly passed away in February 2010 before this paper could be published.

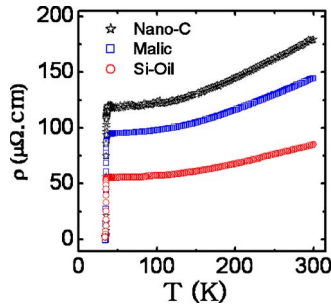


FIG. 1. (Color online) The electrical resistivity  $\rho$  vs temperature  $T$  for the 10 wt % malic acid doped and the 5 wt % nanocarbon doped sample sintered at 800 °C. For comparison, data from Ref. 10 for the 10 wt % silicone oil doped sample sintered at 600 °C are also shown.

such as silicone oil and other oxide or nonoxide nanomaterials.<sup>5–11,19,20</sup> Improvement of the  $j_c(B)$  was quite successful for most of chemical doped  $\text{MgB}_2$ . However, the origin of the pinning mechanisms are still not well understood except the silicon oil doped case.<sup>10</sup>

In this paper, the effects of two kinds of carbon sources, in the form of nanocarbon and malic acid doped  $\text{MgB}_2$  samples were studied. The origin of the improvement of  $j_c(B)$  and vortex pinning mechanism for above cases are analyzed in the framework of the collective pinning theory and compared with the silicon oil doped case. Compare to the  $\delta T_c$  pinning for the pure  $\text{MgB}_2$  over the whole temperature range below  $T_c$ , both  $\delta l$  and  $\delta T_c$  pinning coexist in the  $\text{MgB}_2$  doped with above two kinds of carbon sources.

## II. EXPERIMENTAL

The standard solid state powder processing technique was adapted to prepare the 10 wt % malic acid and 5 wt % nano-C added polycrystalline  $\text{MgB}_2$  samples after the sintering at temperature of 800 °C as described elsewhere.<sup>21</sup> The critical temperature  $T_c$  was defined as the onset temperature at which diamagnetic properties were observed. This temperature was  $35 \pm 0.20$  K for the both samples. X-ray diffraction results revealed that all samples were crystallized in the  $\text{MgB}_2$  structure as the major phase. However, a few impurity lines of  $\text{MgO}$  in the both samples were observed. The resistivity measurements were carried out on the bare cylindrical bar samples by using a physical properties measurement system (PPMS, Quantum Design) in the field range from 0–8.7 T. The magnetic hysteresis loops were measured using a magnetic property measurement system (MPMS, Quantum Design). By using the Bean's critical state model, the critical current density was calculated.

## III. RESULTS AND DISCUSSIONS

The temperature dependencies of the resistivity ( $\rho$ ) for the  $\text{MgB}_2$  samples with different dopants at zero external magnetic fields are shown in Fig. 1. For comparison, data from Ref. 10 for silicone oil doped  $\text{MgB}_2$  with similar  $T_c$  are also shown. It can be seen that the resistivity depends on the particular dopant. Table I presents the measured  $\rho$  values, the residual resistivity ratio,  $RRR$  ( $\rho_{300\text{ K}}/\rho_{40\text{ K}}$ ), and the active cross-sectional area fraction ( $A_F$ ) of  $\text{MgB}_2$  samples for three

TABLE I. The resistivity at 40 and 300 K,  $\Delta\rho = \rho_{300\text{ K}} - \rho_{40\text{ K}}$  and the connectivity factor  $A_F$  of 5 wt % nanocarbon, 10 wt % malic acid, and 10 wt % silicone oil doped  $\text{MgB}_2$ . The resistivity data for the silicone oil doped  $\text{MgB}_2$  sample are extracted from Ref. 10.

Sample	$\rho_{40\text{ K}}$ ( $\mu\Omega\text{ cm}$ )	$\rho_{300\text{ K}}$ ( $\mu\Omega\text{ cm}$ )	$\Delta\rho$ ( $\mu\Omega\text{ cm}$ )	$A_F$
Nanocarbon	119.4	180.4	61.0	0.111
Malic acid	94.9	145.2	50.3	0.145
Silicone oil	55.5	85.3	29.8	0.249

different dopants. The results show that  $\rho$  in the normal state for silicone oil doped  $\text{MgB}_2$  is lower than for the nanocarbon and malic acid doped superconductors. Therefore, the scattering processes are different in these samples.

The connectivity factor,  $A_F$ , from the relation  $A_F = \Delta\rho_{sc}/\Delta\rho$ , where  $\Delta\rho_{sc}$  is the single crystal resistivity and  $\Delta\rho = \rho(300\text{ K}) - \rho(40\text{ K})$ , from our experimentally measured resistivity, was also evaluated by the Rowell analysis.<sup>22</sup> We observed that  $A_F$  of the silicone oil doped sample was larger than that of the malic acid doped sample, as well as the nanocarbon doped sample. As can be seen in Table I,  $A_F$  values ranged from as low as 0.111 (nano-C doped  $\text{MgB}_2$ ) to as high as 0.249 (Si-doped  $\text{MgB}_2$ ), depending on the dopant. However, the  $A_F$  values of all samples are still lower than the ideal value of 1. This indicates that there are some obstacles to current, such as voids or the impurities  $\text{MgO}$  for all samples.

The  $j_c(B, T)$  results are shown in a double-logarithmic plot in Fig. 2 at the temperatures of 20 and 30 K for all samples. The published data for the silicone oil doped  $\text{MgB}_2$  (Ref. 10) is added in Fig. 2. The  $j_c(B, T)$  values for the nanocarbon and malic acid doped  $\text{MgB}_2$  are almost same, while the silicone oil doped  $\text{MgB}_2$  has a larger  $j_c$ . For all the samples, the  $j_c$  initially shows a plateau at low field and then begins to decrease quickly once the field reaches the cross-over field from the single vortex to the small bundle pinning regime,  $B_{sb}$ , which decreases with increasing temperature. Further increasing the field results in a faster drop in  $j_c$  near the irreversibility line, which is obtained by using the criterion of  $j_c = 100\text{ A/cm}^2$ .

The normalized temperature dependence of  $B_{c2}$ , which was obtained from the 90% values of their corresponding resistivity transitions and of  $B_{irr}$  are shown in Fig. 3. The results show that  $B_{c2}$  depends on the dopant while  $B_{irr}$  does

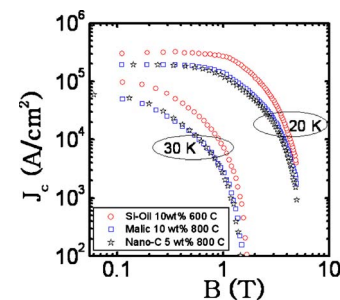


FIG. 2. (Color online) Magnetic dependence of the critical current density  $j_c$  for malic acid and nanocarbon doped  $\text{MgB}_2$ . Results for  $j_c$  of silicone oil doped  $\text{MgB}_2$  (Ref. 10) are also shown.

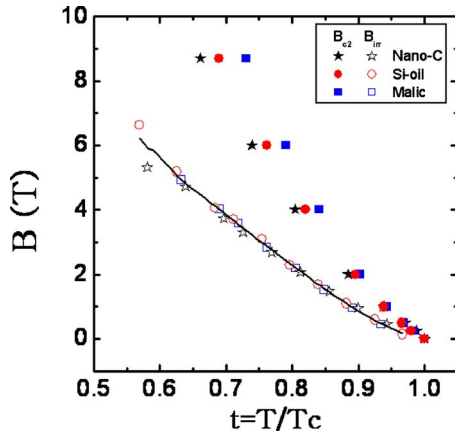


FIG. 3. (Color online) The upper critical and irreversibility fields of 10 wt % malic acid, 5 wt % nanocarbon, and 10 wt % silicone oil doped MgB<sub>2</sub> as a function of temperature. The solid curves are fits to  $B_{irr} = B_{irr}(0)[1 - (T/T_c)^2]^{3/2}$ , with fitting parameter  $B_{irr}(0)$ . Data for silicone oil doped MgB<sub>2</sub> sample have been taken from Ref. 10.

not. The solid curves in Fig. 3 are in good agreement with the typical behavior expected for the case of giant flux creep, which is predicted to be  $B_{irr} \approx (1 - t^2)^{3/2}$ , where  $t = T/T_c$ .<sup>23</sup>

The collective pinning theory which was derived by Blatter *et al.*,<sup>23</sup> shows that critical current density is field independent if the applied magnetic field is lower than the crossover field  $B_{sb}$ . In this low field limit, the interaction between the vortices is negligible and a single vortex pinning mechanism dominates with following relation:

$$B_{sb} \propto j_{sv} B_{c2}, \quad (1)$$

where  $j_{sv}$  is the critical current density. At higher fields, for  $B > B_{sb}$ ,  $j_c(B)$  decreases quickly as field is increased and critical current density follows an exponential form:

$$j_c(B) \approx j_c(0) \exp[-(B/B_0)^{3/2}], \quad (2)$$

where  $B_0$  is a parameter of the order of  $B_{sb}$ .

Whether experimental results  $j_c(B)$  as shown in Fig. 2 follows the Eq. (2) or not,  $-\log[j_c(B)/j_c(0)]$  as a function of  $B$  is drawn in a double-logarithmic plot in the inset of Fig. 4. It is clear that Eq. (2) describes the experimental data quite well for intermediate fields, while deviations from the fitting curves are observed at both low and high fields. The deviation

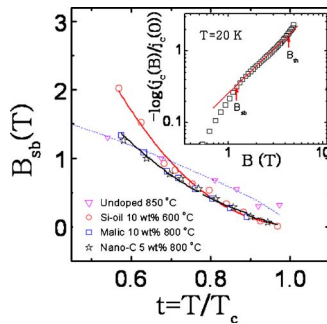


FIG. 4. (Color online) Normalized temperature dependence of the crossover field  $B_{sb}$ . The solid curves are fits to Eq. (4). Inset: double logarithmic plot of  $-\log[j_c(B)/j_c(0)]$  as a function of  $B$ , with the solid line showing the fit given by Eq. (2). The crossover fields  $B_{sb}$  and  $B_{th}$  are shown by arrows. The data for the silicone-oil doped and undoped samples are extracted from Refs. 10 and 24, respectively.

tion at low fields, below  $B_{sb}$ , is from the single vortex pinning regime to the small bundle pinning regime. The deviation at high field is located near to the irreversibility line, which is related to thermal fluctuation effect. The field of this deviation is indicated as  $B_{th}$ .

The crossover field  $B_{sb}$  while varying temperature is drawn in Fig. 4 for pristine MgB<sub>2</sub> sintered at 850 °C, the 10 wt % malic acid doped, and the 5 wt % nanocarbon doped samples. For the comparison, we added data of 10 wt % silicone oil doped, from our previous publication.<sup>10</sup> The results show that the silicone oil (liquid precursor) doped MgB<sub>2</sub> has a larger  $B_{sb}$  value than that of the malic acid and the nanocarbon doped MgB<sub>2</sub>. These results show that the liquid precursor, silicone oil, which produces Si and C on the atomic scale, enhance the connectivity and further improve the  $j_c$  and the crossover field  $B_{sb}$  of MgB<sub>2</sub>.

It is known that the  $\delta T_c$  and  $\delta l$  pinning show different temperature dependencies of the critical current density  $j_{sv}$  in the single vortex pinning regime as  $j_{sv} \propto (1 - t^2)^{7/6} (1 + t^2)^{5/6}$ , with  $t = T/T_c$  and for the case of  $\delta T_c$  pinning, while for  $\delta l$  pinning,  $j_{sv} \propto (1 - t^2)^{5/2} (1 + t^2)^{-1/2}$ .<sup>17,18</sup> Unified version of  $B_{sb}$  is as following:

$$B_{sb}(T) = B_{sb}(0) \left( \frac{1 - t^2}{1 + t^2} \right)^v, \quad (3)$$

where  $v = 2/3$  and 2 for  $\delta T_c$  and  $\delta l$  pinning, respectively.

To find out how much of the  $\delta T_c$  and  $\delta l$  pinning contribute the total pinning mechanism of the doped MgB<sub>2</sub> samples, we separated the  $B_{sb}$  data into two part as follows:

$$B_{sb} = P_1 B_{sb}^{T_c} + P_2 B_{sb}^l, \quad (4)$$

in which  $B_{sb}^{T_c}$  and  $B_{sb}^l$  are the  $B_{sb}$  for of  $\delta T_c$  and  $\delta l$  pinning, respectively. The values of  $P_1$  and  $P_2$  are fitting parameters of  $\delta T_c$  and  $\delta l$  pinning. In order to compare the effects of the  $\delta T_c$  and the  $\delta l$  pinning mechanisms, the  $P$  parameter was defined  $P_{T_c} = P_1 B_{sb}^{T_c} / B_{sb}$  and  $P_l = P_2 B_{sb}^l / B_{sb}$ , which represent  $\delta T_c$  or  $\delta l$  pinning effects, respectively. The  $B_{sb}$  data obtained from inset of Fig. 4 follows Eq. (4) very well as shown in the solid curves in Fig. 4.

From Fig. 4, one can immediately notice that the  $B_{sb}$  of pure MgB<sub>2</sub> is quite different from those of the above three types of nanoparticles doped MgB<sub>2</sub>. The contributions of each pinning effect are shown in Fig. 5. For both doped MgB<sub>2</sub>, both  $\delta T_c$  pinning and  $\delta l$  pinning exist but which pinning mechanism is dominant depends on the temperature range and dopants. In particular,  $\delta l$  pinning is the dominant mechanism at low temperatures but with increasing temperature  $\delta T_c$  pinning becomes dominant. From Fig. 5, one can clearly see the change from the  $\delta l$  to  $\delta T_c$  pinning mechanism at  $t \approx 0.7$ . The contributions of each pinning for the malic acid and nano-C doped MgB<sub>2</sub> are compared with the silicone oil doped MgB<sub>2</sub> (data extracted from the Ref. 10) and the undoped MgB<sub>2</sub> from the Ref. 24, as shown in the Fig. 6. The results clearly show that the  $\delta T_c$  pinning is dominant over the whole temperature range for the pristine MgB<sub>2</sub>.<sup>16</sup> For three doped MgB<sub>2</sub> samples, the dominating pinning mechanism depends on the temperature range and dopants. The temperature  $T_{eq}$  where both pinning mechanisms have equal contributions depends on the dopant as well. The  $T_{eq}$  values



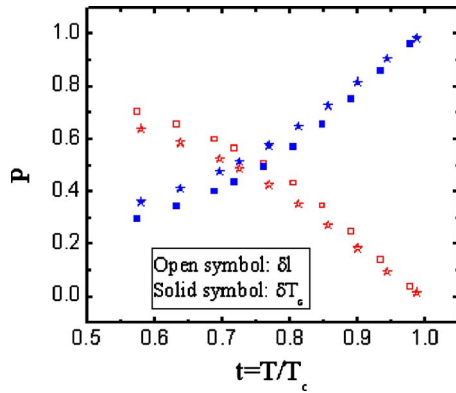


FIG. 5. (Color online)  $\delta l$  and  $\delta T_c$  pinning contributions as a function of normalized temperature for 10 wt % malic acid doped (squares) and 5 wt % nanocarbon doped  $\text{MgB}_2$  sintered at 800 °C (stars).

are 32.5, 26.5, and 24.6 K for silicone oil,<sup>10</sup> malic acid, and nanocarbon doped  $\text{MgB}_2$  samples, respectively. These results suggest that the  $\delta l$  is the dominant for both nanocarbon and malic acid doped  $\text{MgB}_2$  for  $t \approx 0.7$ , while for the silicone oil doped sample this pinning is dominant up to the temperatures close to the  $T_c$ .

In conclusion, we have found that  $\text{MgB}_2$  doped with a liquid precursor, silicone oil,<sup>10</sup> in  $\text{MgB}_2$  results show higher grain connectivity and larger  $j_c(B)$  compared to nonliquid carbon sources of malic acid and nano-C doped  $\text{MgB}_2$  even though this nonliquid carbon sources gave high improvement of  $j_c(B)$  compare to the pristine  $\text{MgB}_2$ . Our results show that the  $\delta l$  pinning mechanism, due to spatial fluctuations of the charge-carrier mean free path, is dominant at low tempera-

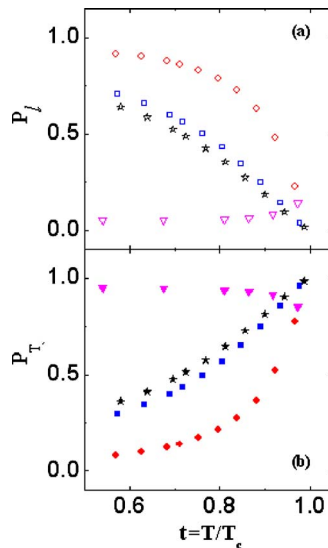


FIG. 6. (Color online) (a)  $\delta l$  pinning and (b)  $\delta T_c$  pinning contributions as a function of normalized temperature for undoped  $\text{MgB}_2$  sintered at 850 °C (triangles), 10 wt % malic acid doped (squares), 5 wt % nanocarbon doped  $\text{MgB}_2$  sintered at 800 °C (stars), and 10 wt % silicone oil doped  $\text{MgB}_2$  sintered at 600 °C (circles) from Ref. 10.

tures in  $\text{MgB}_2$  doped with malic acid, nanocarbon, and silicone oil,<sup>10</sup> while at high temperatures close to  $T_c$ ,  $\delta T_c$  pinning is the dominant pinning mechanism. We found that the increase in the critical current density for the malic acid and nano-C doped  $\text{MgB}_2$  is very much related to the change of the doping mechanism from  $\delta T_c$  to  $\delta l$  pinning and the increase in  $B_{sb}(T)$ .

## ACKNOWLEDGMENTS

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